(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 830 866 B1

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
11.12.2002 Bulletin 2002/50

(51) Int Cl.[7]: **A61L 17/00**

(21) Application number: **97307312.5**

(22) Date of filing: **19.09.1997**

(54) **Process for manufacturing sutures from copolymers of glycolide and epsilon-caprolactone**

Verfahren zur Herstellung von Nahtmaterialien aus Copolymeren aus Glykolid und epsilon-Caprolacton

Procédé de fabrication de sutures en copolymères de glycolide et d'epsilon-caprolactone

(84) Designated Contracting States:
AT DE ES FR GB IT

(30) Priority: **20.09.1996 US 710613**

(43) Date of publication of application:
25.03.1998 Bulletin 1998/13

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Printed by Jouve, 75001 PARIS (FR)

Description

FIELD OF THE INVENTION

[0001] This invention relates to the field of suture manufacture and more specifically it relates to a process for making sutures from copolymers of glycolide and ε-caprolactone.

BACKGROUND OF THE INVENTION

[0002] Copolymers of glycolide and caprolactone have been described by Jamiolkowski et al. in U.S. Patent Nos. 4,605,730 and 4,700,704 and by Bezwada et al. in U.S. Patent No. 5,133,739 (all hereby incorporated by reference). As disclosed by Jamiolkowski and Bezwada, these copolymers may be processed into monofilament sutures which are exceptionally compliant while retaining significant straight tensile strength and knot strength. Jamiolkowski discloses that these glycolide/ε-caprolactone copolymers may be made into sutures by extruding the copolymer into a filament then orienting (stretching or drawing) the filament in a two-step process and annealing the filament for 5 to 16 hours. The fibers disclosed by Jamiolkowski with the lowest Young's Modulus values (under 200 Kpsi) would be expected to be the most compliant fibers.

[0003] Bezwada et al. also discloses glycolide/ε-caprolactone copolymers which are produced by reacting a prepolymer of ε-caprolactone and glycolide with additional glycolide.

[0004] Bezwada describes these copolymer as having very low Young's Modulus values and desirable strength and knot tensile strengths.

[0005] However, it would be desirable to optimize the process used for manufacturing sutures from these copolymers with high straight and knot tensile strengths and low Young's Modulus values.

SUMMARY OF THE INVENTION

[0006] We have discovered a process for producing a suture comprising the steps of (a) extruding melted copolymer composed substantially of glycoside and ε-caprolactone resin through an orifice and rapidly quenching the melted copolymer resin to produce a filament; (b) drawing the filament in the range of from 4X to 7.5X in a first drawing zone to produce a drawn filament; (c) drawing the singly drawn filament in a second drawing zone in the range of from 1X to 4X while in a first heated zone being maintained at a temperature in the range of from 65.6°C (150°F) to 232.2°C (450°F), to form a doubly drawn filament; (d) in-line relaxation of the doubly drawn filament in the range of from 0.65X to 0.98X, in a second heated zone being maintained at a temperature in the range of from 37.8°C (100°F) to 204.4°C (400°F), to form a relaxed filament; then rack annealing the relaxed filament.

[0007] Preferably, the second heated zone is maintained at a temperature in the range of from 50°C (122°F) to 148.9°C (300°F).

BRIEF DESCRIPTION OF THE FIGURES

[0008] The FIGURE is a side elevation, partially schematic of an apparatus suitable for carrying out the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0009] As used herein the term glycolide/ε-caprolactone copolymers shall include copolymers composed primarily of repeating units having the chemical formula I and II:

$$[\text{-O-(CH}_2)_5\text{-C(O)-}]_A \qquad \text{I}$$

$$[\text{-O-(CH}_2)\text{-C(O)-}]_B \qquad \text{II}$$

[0010] Preferred are the glycolide/ε-caprolactone copolymers described in U.S. Patent Nos. 4,605,730; 4,700,704 and 5,133,739. However, minor amounts (i.e. less than 10% by weight and preferably less than 5% by weight) of other aliphatic ester monomers may also be present in these copolymers. Suitable additional repeating units include [-O-R_5-C(O)-] where R_5 is selected from the group consisting of -C(R_6)(R_7)-, -$(CH_2)_3$-O-, CH_2-CH_2-O-CH_2-, -CR_8H-CH_2-,

$-(CH_2)_F-O-C(O)-$ and $-(CH_2)_F-C(O)-CH_2-$; R_6 and R_7 are independently hydrogen or an alkyl containing from 1 to 8 carbon atoms, provided that R_6 and R_7 are not both hydrogen; R_8 is hydrogen or methyl and F is an integer in the range of from 2 to 6. Suitable aliphatic esters repeating units include but are not limited to repeating units selected from the group consisting of p-dioxanone, trimethylene carbonate, lactide, 1,5-dioxepan-2-one, 1,4-dioxepan-2-one and 6,6-dimethyl -1,4-dioxepan-2-one.

[0011] The preferred method for preparing the flexible glycolide/ε-caprolactone sutures of the present invention utilizes as the raw material pellets of glycolide/ε-caprolactone copolymer (prepared as described by Bezwada) having a weight average molecular weight of from 59,000 MW to 100,000 MW and has a crystallinity of greater than 10 percent and preferably greater than 15 percent as measured by x-ray diffraction.

[0012] Referring to the FIGURE, there is shown an apparatus that is suitable for carrying out the present invention. An extruder 10 is terminated at one end with an extrusion die 12. A longitudinal extruder screw is mounted for rotation within the barrel 16 and is driven by a variable speed motor 18 through a gear 20. Polymer pellets are introduced into the extruder through hopper 22 which communicates with the barrel 16. In normal operation of the extruder 10, the feeding zone 24 of the extruder is maintained at a temperature in the range of from 176.7°C (350°F) to 232.2°C (450°F), the transition zone 26 is maintained at a temperature in the range of from 176.7°C (350°F) to 232.2°C (450°F), and the pump block 30, block 28 and die 12 are maintained at a temperature in the range of from 171.1°C (340°F) to 260°C (500°F). A pump 33 driven by a motor 32, pumps the molten copolymer through spinneret orifices in the die 12 to form a plurality of filaments 31 (for simplicity only one filament is shown in the FIGURE). The filament 31 is extruded into quench bath 34. The quench bath 34 is filled with a liquid heat exchange medium. The surface of the liquid in the quench bath 34 is preferably not more than a few centimeter below the die 12 in order to achieve rapid cooling of the extruded filament 31. Preferably, the gap will be in the range of from 0.635 cm (1/4") to 17.78 cm (7"). The quench bath 34 is maintained at a temperature below 48.9°C (120°F) and preferably the quench bath 34 is maintained at about room temperature. The filament 31 enters the quench bath 34 and travels around idler roll 36 in the quench bath 34 and then up out of the quench bath 34 to another idle roller 35 then to the first godet 37 in the first drawing zone 2. In the first drawing zone 2 the filament 31 is drawn in the range of from 4X to 7.5X its original length. The filament 31 may be drawn incrementally or in several discrete steps in the first drawing zone 2. The drawing may preferably be performed in a heated cabinet, or by using heated godets).

[0013] In the preferred embodiment of the invention shown in the attached Figure, the filament 31 is drawn by a first godet 37 and a second godet 42. The first godet 37 includes several rolls 38. The first godet 37 is rotated at a peripheral speed that is equal to or slightly higher than the speed at which the filament 31 is extruded from the die orifice 12. The first godet 37 may be combined with a nip roller (not shown) to assure the filament 31 does not slip in the subsequent drawing to the extruded filament 31. The first draw of the extruded filament 31 will be performed by feeding the extruded filament 31 from the first godet 37 to second godet 42 which includes several rolls 43. The second godet 42 is rotated at a peripheral speed that is in the range of from about 4X to about 7.5X of the speed of the first godet 37.

[0014] The filament 31 then passes into a second drawing zone 4, where the filament 31 is drawn again in the range of from 1X to 4X while in a first heated zone 46. The filament 31 may be drawn incrementally or in one or more discrete steps in the second drawing zone 4. The drawing will be performed in a first heated zone 46. The temperature of the first heated zone 46 will be in the range of from 65.6°C (150°F) to 232.2°C (450°F), preferably in the range of from 79.4°C (175°F) to 204.4°C (400°F). The filament 31 will remain in the first heated zone 46 generally only a short time preferably in the range of from 1.0 seconds to 30 seconds.

[0015] In the preferred embodiment of the invention shown in the attached Figure, the filament 31 passes through a first heated zone 46 to a third godet 50. The first heated zone 46 is preferably an orienting oven 48. The filament 31 is drawn in the range of from 1X to 4X, while traveling from the second godet 42 to the third godet 50 in the first heated zone 46. The third godet 50 includes a main roll 51 and an air bearing 52, that are rotating at a peripheral speed of 1X to 4X of the peripheral speed of the second godet 42. Preferably the draw ratio will be in the range of from 1X to 3X, preferably 1.0X to 1.9X.

[0016] The filament 31 then passes from the second drawing zone 4 into an annealing zone 6, where the filament 31 is annealed and allowed to shrink. In the annealing zone 6 the filament 31 is placed in a second heated zone 54 that is maintained at a temperature in the range of from 37.8°C (100°F) to 204.4°C (400°F), preferably from 65.6°C (150°F) to 176.7°C (350°F), wherein the filament is allowed to shrink to the range of from 98 percent to 65 percent, preferably from 98 percent to 75 percent and more preferably from 95 to 75 percent of the filament original length. The filament 31 may be allowed to shrink incrementally or in one or more discrete steps in the second heated zone 54. The filament 31 will remain in the second heated zone 54 for a short time generally in the range of from 1.0 to 30 seconds and preferably in the range of from 3.0 seconds to 20 seconds.

[0017] In the preferred embodiment of the present invention shown in the attached FIGURE, the filament 31 passes through a second heated zone 54 to a fourth godet 56. The heated zone 54 is preferably an annealing oven 60. The fourth godet 56 includes several rolls 57 that are rotating at a peripheral speed of 0.8X to about 0.98X of the peripheral speed of the third godet 50. Preferably the relaxation ratio will be in the range of from 0.75 to 0.9X. After passing around

the fourth godet 56, the filament 31 is then wound on a spool 70 and transferred to a creel or rack for additional annealing.

[0018] The overall draw ratio, that is, the difference between the peripheral speed of the fourth godet 56 and the first godet 37, will ordinarily be from 6X to 8X and preferably the total draw ratio will be in the range of from 6.9X to 7.2X.

[0019] The residence time of filament 31 within any of the heated zones can be optimized to improve fiber properties. The overall residence time that filament 31 is present in the first and second heated zones will preferably be in the range of from 2 seconds to 50 seconds and most preferably in the range of from 4 seconds to 30 seconds. The residence time can be increased with longer ovens or by having multiple wraps of the fiber in the oven.

[0020] Suitable creels or racks for annealing filament 31 have been described in the art such as the creels disclosed by Listner et al. in U.S. Patent No. 3,630,205. However, unlike the creels disclosed by Listner which permit the filaments to contract as they are annealed in the present invention, it is preferred to fix both ends of the creel and anneal the filaments with no relaxation. Once wrapped on the creel the filament should be annealed in an oven at a temperature of in the range of from 85°C to from 125°C. The filaments should be annealed for in range of from 4 hours to 8 hours, preferably from 5 hours to 7 hours. The filaments may be removed from the creel by cutting the filaments at opposite ends of the creel. The filaments may be attached to needles, packaged and sterilized (by ethylene oxide or other appropriate techniques).

[0021] The following non-limiting examples are further provided to illustrate the practice of the present invention.

EXAMPLE

[0022] A dyed 75/25 weight percent glycolide/caprolactone segmented block copolymer made as described in U.S. Patent No. 5,133,739 was used to produce surgical sutures under the conditions set forth in Table 1 below.

[0023] The polymer was generally made by adding into a dry 56.78L (15 gallon) reactor provided with agitator and oil circulating jacket 7406 grams (64.9 moles) of ε-caprolactone, 9205 grams (79.3 moles of glycolide), 19.71 ml. (0.207 moles) of diethylene glycol (DEG) and 13.68 ml. (0.0045 moles) of stannous octoate (0.33 molar solution in toluene). The reactor contents are evacuated and the vacuum is released with nitrogen. The evacuation and vacuum release cycle is repeated once more, each cycle lasting approximately 25 minutes. The circulating heating oil temperature is set at 195°C. and the batch temperature is monitored. This first stage polymerization reaction is allowed to proceed for 6 hours measured from the time that the batch temperature reaches 190°C. The oil temperature is increased to 216°C. and 13389 grams (115.4 moles) of molten glycolide is added from a melt tank with agitation. After 10 minutes the oil temperature is reset to 204°C. Approximately 60 minutes after the addition of the molten glycolide, the batch temperature begins to be greater than the oil temperature. This point is referred to as the crossover point (XO) and is selected as the"zero time" for the second stage exothermic reaction. At 70 minutes from zero time, the bottom gate of the reactor is opened, the oil is set at 212°C. and the polymer is forced into the pelletizer system. The reactor contents are discharged in 25 minutes.

[0024] The polymer is then pelletized using a cutter speed maintained in the range of 3000 to 3100 RPM. A four blade cutter is used. The die holes are 0.2794 cm (0.11") in diameter and 12 open holes are used. The tempering water recirculation rate is 3.7854 L/s (60 gallons/minute) and the water is maintained at 13°C. The copolymer pellets are separated from the water by means of a centrifugal dryer. The pellets are dried in a vacuum tumble dryer provided with a heating jacket. The drying cycle is 18 hours at room temperature followed by 24 hours at 110°C. The copolymer had an inherent viscosity of 1.66 dl/g measured in hexafluoroisopropanol. The molecular weight by Gel Permeation Chromatography was MW=82,000 daltons. The melting point was 214°C. The composition determined by NMR was 24.5 mole % polycaprolactone (PCL) and 74.7 mole % polyglycolic acid (PGA). The polymers made in this manner were then extruded into filaments under the following conditions:

TABLE 1

Sample No.	1 Reference	2	3	4	5	6
Polymer IV	1.85	1.85	1.85	1.85	1.85	1.85
Filament Size	1	1	1	1	1	1
Feed Zone °C °F	204.4 400	204.4 400	204.4 400	204.4 400	204.4 400	204.4 400
Transition Zone °C °F	204.4 400	204.4 400	204.4 400	204.4 400	204.4 400	204.4 400
Pump °C °F	243.3 470	243.3 470	243.3 470	243.3 470	243.3 470	243.3 470

TABLE 1 (continued)

Sample No.	1 Reference	2	3	4	5	6
Block °C °F	243.3 470	243.3 470	243.3 470	243.3 470	243.3 470	243.3 470
Die °C °F	248.9 480	243.3 470	248.9 480	243.3 470	243.3 470	243.3 470
Barrel kPa PSI	10636 1503	10377 1505	10363 1503	10356 1502	10356 1502	10356 1502
Pump kPa PSI	16485 2391	14079 2042	17458 2532	19671 2853	19671 2853	19671 2853
Die kPa PSI	2586 375	2758 400	2882 418	3192 463	3192 463	3192 463
Pump RPM	6.6	6.8	7.0	6.9	6.9	6.9
Air Gap inches cm	5.08 2	5.08 2	5.08 2	5.08 2	5.08 2	5.08 2
Quench Bath °C °F	20 68	20 68	20 68	20 68	20 68	20 68
Screw RPM	6.5	6.6	6.7	6.6	6.6	6.6
Godet 1cm/s/°C FPM/°F	14.63 16	14.63 16	14.63 16	14.63 16	14.63 16	14.63 16
Godet 2cm/s/°C FPM/°F	77.69 85	77.69 85	77.69 85	77.69 85	77.69 85	77.69 85
Orienting °C Oven °F	176.7 350	176.7 350	176.7 350	176.7 400	176.7 400	176.7 400
Godet 3cm/s/°C FPM/°F	100.54 110	100.54 110	105.11 115	105.11 115	105.11 115	105.11 115
Annealing °C Oven °F	-	121.1 250	121.1 250	79.4 175	79.4 175	79.4 175
Godet 4cm/s/°C FPM/°F	-	89.57 98	95.06 104	95.06 104	95.06 104	95.06 104
Total Draw Ratio	6.88	6.88	7.19	7.19	7.19	7.19
% of In-Line Relaxation	N/A	10%	10%	10%	10%	10%
RPM is revolutions per minute. FPM is feet per minute.						

[0025] Extruded filaments 1-6 were wound on racks and annealed. Sample I, which is a reference sample, was wound on a rack which allowed the filaments to shrink 10% during the annealing process. Samples 2-5 were wound on racks which did not allow the filaments to shrink during the annealing process. Samples 14 were annealed at 105°C for six (6) hours. Samples 5 and 6 were annealed at 120°C and 125°C respectively for six (6) hours. All the samples were tested using the following test procedures. The data from these test are presented in Table 2.

[0026] The characteristic properties of samples 1-6 were determined by conventional test procedures. The tensile properties (i.e., straight and knot tensile strengths and elongation) displayed herein were determined with an INSTRON Tensile Tester. The settings used to determine the straight tensile, knot tensile and break elongation were the following, unless indicated:

TABLE 2

	GAUGE LENGTH (cm)	CHART SPEED(cm)	CROSSHEAD SPEED (cm/min.)
STRAIGHT TENSILE	12.7	30.5	30.5
KNOT TENSILE	12.7	30.5	30.5
BREAK ELONGATION	12.7	30.5	30.5

[0027] The straight tensile strength was calculated by dividing the force to break by the initial cross-sectional area of the suture. The elongation at break was read directly from the stress-strain curve of the sample.

[0028] The knot tensile strength of a suture was determined in separate tests. The surgeon's knot was a square knot in which the free end was first passed twice, instead of once, through the loop, and the ends drawn taut so that a single knot was superimposed upon a compound knot. The first knot was started with the left end over the right end and sufficient tension was exerted to tie the knot securely.

[0029] The specimen was placed in the INSTRON Tensile Tester with the knot approximately midway between the clamps. The knot tensile strength was calculated by dividing the force required to break by the initial cross-sectional area of the fiber. The tensile strength values are reported in MPa (Kpsi = psi x 10^3).

TABLE 3
Comparison of In-Line and Rack Annealed Properties

Sample No.	Size	Diameter mm mils	Tensile N lbs	Strength MPa Kpsi	Knot N lbs	Strength Mpa Kpsi	Elongation %	Modulus Mpa Kpsi
1 (reference)	1	0.5321 20.95	154.04 34.63	692.9 100.5	79.67 17.91	358.5 52.0	46.47	742.6 107.7
2	1	0.5311 20.91	161.87 36.39	730.2 105.9	77.53 17.43	350.3 50.8	40.85	811.5 117.7
3	1	0.5349 21.06	174.68 39.27	777.7 112.8	85.05 19.12	378.5 54.9	40.78	766.7 111.2
4	1	0.5311 20.91	177.13 39.82	799.8 116.0	79.93 17.97	360.6 52.3	38.73	843.2 122.3
5	1	0.5298 20.86	167.79 37.72	761.2 110.4	81.54 18.33	369.6 53.6	37.28	661.2 95.9
6	1	0.5297 20.85	167.87 37.74	761.9 110.5	85.32 19.18	387.5 56.2	38.01	713.6 103.5

[0030] Sample Numbers 2-6 were produced by the inventive in-line annealing process described above. Sample 1, which is a reference sample, was produced by rack annealing the sutures following conventional manufacturing procedures. The data above demonstrate that the inventive process produces sutures that have approximately a 10% increase in the tensile strengths without significantly increasing the Young's modulus of the sutures.

[0031] As shown in Table 4 below the BSR profile of the inventive process also improved as compared to the current process used to manufacture sutures from copolymers of glycolide and ε-caprolactone.

TABLE 4

Comparison of Inventive Process and Conventional Process

BSR Profiles

	1	2	3	4	5	6
Sample #	Control	43-2	44-4	46-2A	46-2B	46-2C
BSR						
0 Days N Lbs	154.04 34.63	161.87 36.39	174.68 39.27	177.13 39.82	167.79 37.72	167.88 37.74
7 days N Lbs	81.18 18.25	90.43 20.33	99.15 22.29	105.33 23.68	91.46 20.56	91.86 20.65
Percent strength remaining from initial strength	53%	56%	57%	59%	55%	55%

Claims

1. A process for producing a suture from a copolymer of glycolide and ε-caprolactone comprising the steps of (a) extruding a melted copolymer of glycolide and ε-caprolactone resin through an orifice and rapidly quenching the melted copolymer resin to produce a filament; (b) drawing the filament in the range of from 4X to 7.5X in a first drawing zone to produce a drawn filament; (c) drawing the singly drawn filament in a second drawing zone in the range of from 1X to 4X while in a first heated zone being maintained at a temperature in the range of from 65.6°C (150°F) to 232.2°C (450°F), to form a doubly drawn filament; (d) in-line relaxation of the doubly drawn filament in the range of from 0.65X to 0.98X, in a second heated zone being maintained at a temperature in the range of from 37.8°C (100°F) to 204.4°C (400°F), to form a relaxed filament; then rack annealing the relaxed filament to form a glycolide/ε-caprolactone suture.

2. The process of claim 1 wherein the filament in the second drawing zone is exposed to a first heated zone being maintained at a temperature in the range of from 79.4°C (175°F) to 204.4°C (400°F).

3. The process of claim 1 wherein the filament is drawn in a single step in the first draw zone.

4. The process of claim 2 wherein the filament is drawn by a second and a third godets.

5. The process of claim 4 wherein the doubly drawn filament is maintained second heated zone in the range of from 3 seconds to 20 seconds.

6. The process of claim 4 wherein the singly drawn filament is drawn in the range of from 1X to 3X the second drawing zone.

7. The process of claim 1, wherein: in step (c) the singly drawn filament is drawn in a second drawing zone in the range of from 1.0X to 1.9X; the second heated zone is maintained at a temperature in the range of from 65.6°C (150°F) to 176.7°C (350°F); and the rack annealing of the relaxed filament is at a temperature of from 85°C to 125°C for at least 4 to 8 hours.

8. The process of claim 7 wherein the total draw ratio of the suture from drawing and relaxing is in the range of from 6 to 8.

9. The process of claim 7 wherein the relaxed filament is annealed for in the range of from 5 hours to 7 hours.

10. A suture obtainable by the process of any one of claims 1 to 9.

Patentansprüche

1. Verfahren zum Herstellen eines Nahtmaterials aus einem Copolymer aus Glycolid und ε-Caprolacton, das die Schritte umfaßt (a) Extrudieren eines geschmolzenen Copolymers aus Glycolid und ε-Caprolacton, durch eine Düse und schnelles Abkühlen des geschmolzenen Copolymerharzes, um ein Filament herzustellen; (b) Ausziehen des Filaments im Bereich von 4X bis 7,5X in einem ersten Ausziehbereich, um ein ausgezogenes Filament herzustellen; (c) Ausziehen des einzeln-ausgezogenen Filaments in einem zweiten Ausziehbereich im Bereich von 1X bis 4X, während ein zuerst erhitzter Bereich auf einer Temperatur im Bereich von 65,6°C (150° F) bis 232,2°C (450°F) gehalten wird, um ein doppelt-ausgezogenes Filament zu bilden; (d) lineare Entspannung des doppelt-ausgezogenen Filaments im Bereich von 0,65X bis 0,98X, wobei eine zweite erhitzte Zone auf einer Temperatur im Bereich von 37,8°C (100°F) bis 204,4°C (400°F) gehalten wird, um ein entspanntes Filament zu bilden; dann "rack"-Härtung des entspannten Filaments, um ein Glycoid/ε-Caprolactonnahtmaterial zu bilden.

2. Verfahren nach Anspruch 1, wobei das Filament im zweiten Ausziehbereich einer ersten geheizten Zone ausgesetzt ist, die auf einer Temperatur im Bereich von 79,4°C (175°F) bis 204,4°C (400°F) gehalten wird.

3. Verfahren nach Anspruch 1, wobei das Filament in einem einzigen Schritt im ersten Ausziehbereich ausgezogen wird.

4. Verfahren nach Anspruch 2, wobei das Filament durch eine zweite und eine dritte Galette ausgezogen wird.

5. Verfahren nach Anspruch 4, wobei das doppelt-ausgezogene Filament in der zweiten erwärmten Zone im Bereich von 3 Sekunden bis 20 Sekunden gehalten wird.

6. Verfahren nach Anspruch 4, wobei das einzeln-ausgezogene Filament im Bereich von 1X bis 3X im zweiten Ausziehbereich ausgezogen wird.

7. Verfahren nach Anspruch 1, wobei in Schritt (c) das einzeln-ausgezogene Filament in einem zweiten Ausziehbereich im Bereich von 1,0X bis 1,9X ausgezogen wird; die zweite erhitzte Zone auf einer Temperatur im Bereich von 65,6°C (150°F) bis 176,7°C (350°F) gehalten wird; und das "rack"-Härten des entspannten Filaments bei einer Temperatur von 85°C bis 125°C für mindesten 4 bis 8 Stunden stattfindet.

8. Verfahren nach Anspruch 7, wobei das gesamte Auszieh-Verhältnis des Nahtmaterials vom Ausziehen und Entspannen im Bereich von 6 bis 8 liegt.

9. Verfahren nach Anspruch 7, wobei das entspannte Filament für den Bereich von 5 Stunden bis 7 Stunden gehärtet wird.

10. Nahtmaterial, erhältlich nach dem Verfahren nach einem der Ansprüche 1 bis 9.

Revendications

1. Procédé pour la production d'une suture a partir d'un copolymère de glycolide et de ε-caprolactone comprenant les étapes consistant à (a) extruder un copolymère de résine fondue de glycolide et de ε-caprolactone par un orifice et en trempant rapidement la résine de copolymère fondu pour produire un filament ; (b) étirer un filament dans une échelle de 4 fois a 7,5 fois dans un premier banc d'étirage pour produire un filament étiré ; (c) étirer un filament étiré un seule fois dans un second banc d'étirage dans une échelle de 1 fois à 4 fois à 65,6 °C tandis qu'il est dans le premier banc chauffé, maintenu à une température située dans une échelle de 65,6 °C (150 °F) à 232,3 °F (450 °F), pour former un filament doublement étiré ; (d) relaxer en ligne le filament doublement étiré d'un ordre de 0,65 fois à 0,98 fois dans la seconde zone chauffée, en maintenant la température dans une échelle variant de 37,8 °C (100 °F) à 204,4 °C (400 °F), pour former un filament détendu ; et ensuite recuire sur support le filament détendu pour former une suture de glycolide/ε-caprolactone.

2. Procédé selon la revendication 1, dans lequel le filament dans le deuxième banc d'étirage est exposé à la première zone chauffée en étant maintenu à une température dans une échelle de 79,4 °C (175 °F) à 204,4 °C (400 °F).

3. Procédé selon la revendication 1, dans lequel le filament est étiré en une seule étape dans le premier banc d'étirage.

4. Procédé selon la revendication 2, dans lequel le filament est étiré dans une deuxième et une troisième chambre à godets.

5. Procédé selon la revendication 4, dans lequel le filament doublement étiré est maintenu dans une deuxième zone chauffée pour une durée variant de 3 secondes à 20 secondes.

6. Procédé selon la revendication 4, dans lequel le filament étiré une seule fois est étiré dans une échelle variant de 1 fois à 3 fois dans la seconde zone chauffée.

7. Procédé selon la revendication 1, dans lequel :
dans l'étape (c), le filament étiré une fois est étiré dans un deuxième banc d'étirage dans l'ordre de 1,0 fois à 1,9 fois, la seconde zone chauffée étant maintenue à une température située dans une échelle de 65,6 °C (150 °F) à 176,7 °C (350 °F), et le recuit sur support du filament détendu se faisant à une température variant de 85 °C à 125 °C pour au moins 4 à 8 heures.

8. Procédé selon la revendication 7, dans lequel le rapport d'étirage total de la suture de l'étirage et de la relaxation est de l'ordre de 6 à 8.

9. Procédé selon la revendication 7, dans laquelle le filament détendu est recuit pour une durée variant de 5 heures à 7 heures.

10. Suture qui peut être obtenue par le procédé de n'importe laquelle des revendications 1 à 9.

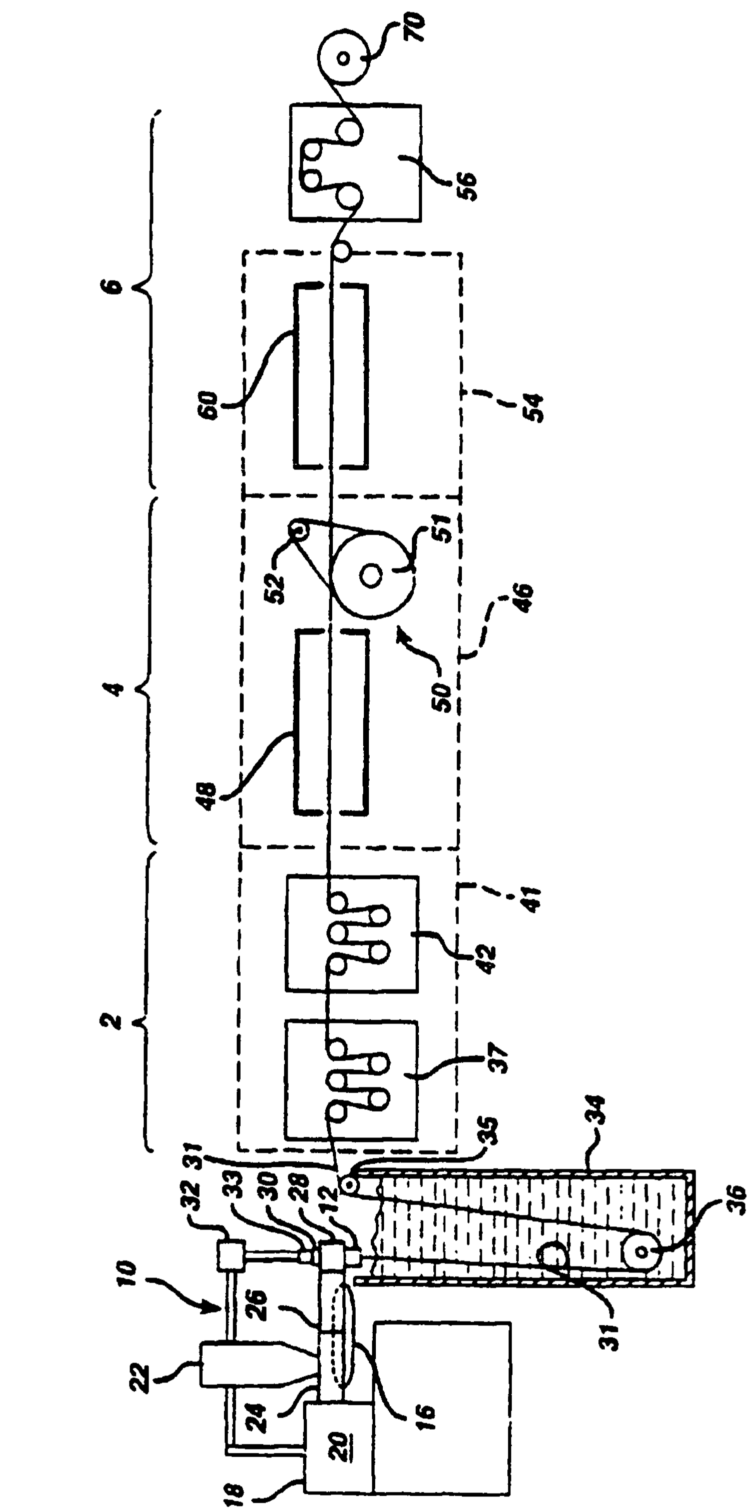
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FIG. 1